Method of Test for

DETERMINATION OF EMBEDMENT COATING ON EMBEDMENT COATED GLASS BEADS FOR PAVEMENT MARKINGS

DOTD Designation: TR 530-10

I. Scope

A. This method describes the presence of embedment coating on glass beads.
B. Reference Documents.
   1. DOTD Sampling Procedure S-608, Traffic Paint and Glass Beads
   2. ASTM D346 – Collection and Preparation of Coke Samples for Laboratory Analysis

II. Apparatus

A. **Graduated Cylinder** – 50mL
B. **Acetone** – Reagent Grade
C. **Dansyl Chloride 98% Purity**
D. **Analytical Balance** – 50g to an accuracy 0.1 g
E. **Brown Glass Container with friction top lid –** 0.5L
F. **Rubber Gloves**
G. **Safety Glasses or Goggles**
H. **Medicine Dropper**
I. **4″ Glass Filter Paper**
J. **Aluminum Weighing Dish** – small
K. **2″ Buchner Funnel and Suction Flask**
L. **2″ Whatman No. 1 Filter Paper or Equivalent**
M. **Vacuum Pump**
N. **Ultraviolet Light Source,** with longwave UV lamps, minimum of 100 watts
O. **Drying Oven –** capable of maintaining a temperature of 60°C ± 5°C.
P. **Glassbead Worksheet (Figures 1 and 2)**
Q. **Spatula**
R. **Acetone Spray Bottle**
S. **Splitter (16:1 and 1:1 ratio)**

III. Reagents

Dansyl Chloride Solution – Weigh 0.2 g of Dansyl Chloride and dissolve it in 25mL of acetone. This solution must be stored in a tight closed container, and it must be refrigerated in the brown glass container with friction top lid. Discard the dansyl chloride solution after 24 hours due to it being unusable after a day. Refer to MSDS sheet for both chemicals for proper disposal.

IV. Health Precautions

Proper equipment and precautions are to be used whenever Dansyl Chloride is handled. Use protective gloves and eye protection. **Do not get on skin**. Proper ventilation may be required while handling dansyl chloride or acetone. Refer to MSDS sheet for both chemicals.

V. Sample

Glassbeads shall be sampled in accordance to DOTD Sampling Procedure S-608.

VI. Procedure

A. Set drying oven to 60 ± 5ºC.
B. Turn on utlraviolet light source.
C. Using the splitter, obtain 2 test specimens of beads approximately 10g each. Test specimens can be obtained by ASTM D346 (which describes and illustrates the quartering procedure for reducing bulk samples in order to get

representative test samples of suitable amount). Retain one test specimen, which shall be your control sample, for fluorescence comparison.

D. Place 2" filter paper into the Buchner funnel and attach to suction flask.
E. Place beads into the Buchner funnel and saturate the specimen with the Dansyl Chloride solution using the medicine dropper.
F. Let solution stand for at least 30 seconds.
G. Place saturated beads into an aluminum dish and dry in oven at 60 ± 5ºC for 15 to 20 minutes. Beads will yellow and may clump).
H. Remove test specimen from oven and place glass beads in Buchner funnel with new filter paper. Spray beads with a spray bottle filled with acetone until the yellow color is removed completely and the beads become clear and transparent. Use suction from vacuum pump throughout this step.
I. Remove beads from the funnel and place into a new aluminum tray.
J. Place the aluminum tray with test specimen to dry in an oven (60°C ± 5°C) for 10 minutes or until beads are completely dry.
K. Remove beads from the oven and place on filter paper. If beads are clumping gently break them apart with a spatula.
L. Inspect the test specimen under the ultra violet light in a completely darkened room.

VII. Observations

A. All embedment coated beads will emit a yellow green fluorescent light.
B. If additional fluorescence is observed when compared to the control sample (Step VI.C.), the lot is accepted. Record a "P" for passing on one of the corresponding glassbead worksheet (Figure 1 or 2).
C. If no fluorescence is observed, the test shall be conducted once more with a new sample. If the second test specimen fails, then record "F" for failing on the corresponding glassbead worksheet (Figure 1 or 2).

VIII. Normal Test Reporting Time

Normal test reporting time is 2 days.

8/02

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Materials & Testing Section

LARGE EMBEDMENT COATED GLASS BEADS
FOR PAINTED TRAFFIC STRIPING

REMARKS 2

	TEST RESULTS (Max. of 15 Characters)	P/F
	xxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxx	xxx
COLOR		
DESCRIPTION		
EMBEDMENT COATING	xxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxx	P
ROUNDNESS, % (DOTD Spec. 157-002)	100	P
ROUNDNESS, % (DOTD Spec. 157-002)		
	xxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxx	xxx
	xxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxx	xxx
ANGULAR PARTICLES, % (DOTD Spec. 157-002)	0	P
	xxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxx	xxx
GRADATION (SIEVE DESIGNATION): (ASTM D 1214)	xxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxx	xxx
RETAINED # 1.70 mm, %	0	P
RETAINED # 1.40 mm, %	0	P
RETAINED # 1.18 mm, %	5	P
RETAINED # 1.00 mm, %	40	P
RETAINED # 0.85 mm, %	10	P
RETAINED # 0.71 mm, %	0	P
PAN, %	0	P

Sample Weight, g. (W_i) _____ Sieve No.	1.70	1.40	1.18	1.00	0.85	0.71	PAN
Sieve + Retained Wt., g.							
Sieve Wt., g.							
Retained Wt., g. (W_x)							
Retained, % $\frac{W_x}{W_a} \times 100$							

Total Retained Wt., g (W_a) = ΣW_x _____

Percent Difference, % = $[(W_i - W_a)/W_i] \times 100$ _____

09/ 061-001-00000 /343/ 22-70000 /N (OVER)

Figure 1a
Glassbeads (For Painted Traffic Striping) Worksheet Front

xxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxx	xxx
REFRACTIVE INDEX . . . . . (DOTD Spec. 157-002 & AASHTO M 247.4.3)	1.52	P

Tested by: W B Date: 1/6/10 Checked by: ZH Date: 1/6/10

APPROVED BY: _____ Date: _____

Figure 1b
Glassbeads (For Painted Traffic Striping) Worksheet Back

12/08

Louisiana Department of Transportation and Development
Materials & Testing Section
GLASS BEADS (TYPE 1 M)
(AASHTO M 247)

REMARKS 2

	TEST RESULTS (Max. of 15 characters)	P/F
	xxxxxxxxxxxxxxxxxxxxxx	xxx
BATCH NUMBER		xxx
DOTD LOT NUMBER		xxx
COLOR		
EMBEDMENT COATING		P
ROUNDNESS	100 %	P
	xxxxxxxxxxxxxxxxxxxxxx	xxx
	xxxxxxxxxxxxxxxxxxxxxx	xxx
	xxxxxxxxxxxxxxxxxxxxxx	xxx
GRADATIONS:		
PASSING 1.18 mm, % (No. 16)	100	P
PASSING 0.850 mm, %(No. 20)	90	P
PASSING 0.600 mm, % (No. 30)	50	P
PASSING 0.425 mm, % (No. 40)	15	P
PASSING 0.300 mm, % (No. 50)	0	P
PASSING 0.180 mm, % (No. 80)	0	P

Sample Mass, g (M_s) 50 grams

Sieve No.	Sieve Mass, g	Sieve + Retained Mass, g	Retained Mass, g	% Retained	% Coarser	% Passing
1.18 mm (No. 16)						
0.850 mm (No. 20)						
0.600 mm (No. 30)						
0.425 mm (No. 40)						
0.300 mm (No. 50)						
0.180 mm (No. 80)						
Pan						

Total Retained Mass, g (M_R) _____

% Difference, % = $\left(\frac{M_s - M_R}{M_s}\right) \times 100$

	xxxxxxxxxxxxxxxxxxxxxx	xxx
	xxxxxxxxxxxxxxxxxxxxxx	xxx
REFRACTIVE INDEX	1.52	P
FLOW CHARACTERISTICS	xxxxxxxxxxxxxxxxxxxxxx	

Tested by: W B Date: 1/6/10 Checked by: [illegible] Date: 1/6/10

APPROVED BY: _____ Date: _____

09/ 061-00-0000 / 674 / 22-7000000 /N

Figure 2
Glassbeads (Type 1M) Worksheet